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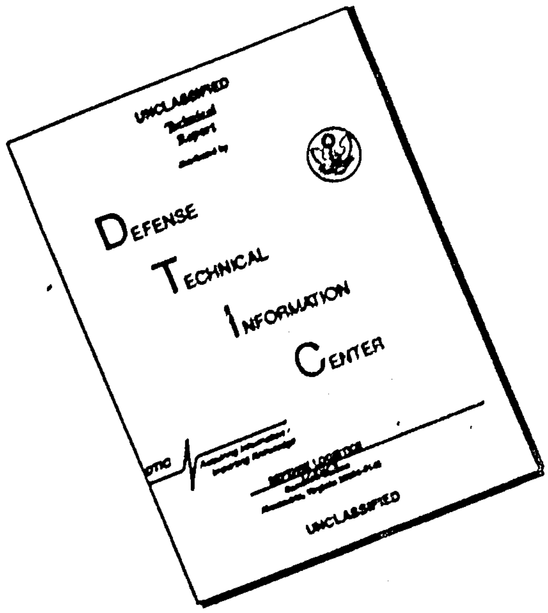

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AFRPL-TR-67-88

March 1967

Special Report

REVIEW AND EVALUATION OF NF SENSITIVITY PROBLEMS (U)

By: M.E. Hill

Air Force Rocket Propulsion Laboratory
Research and Technology Division
Air Force Systems Command
Edwards Air Force Base, California

Contract AF 04(611)-11547

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FOREWORD

This special report was prepared by Stanford Research Institute, Menlo Park, California, under Contract AF 04(611)-11547 for the Air Force Rocket Propulsion Laboratory. It deals with part of a program in which we have surveyed the existing sensitivity information on NF compounds. The program has been the responsibility of the Synthesis Research Department of the Chemical Synthesis and Development Division. We wish to acknowledge the valuable discussions with M.W. Evans, L.B. Seely, A.B. Amster, and S.K. Brauman in developing some of the information and ideas discussed in this report.

The Air Force Rocket Propulsion Laboratory contract monitor is Dr. William Leahy, RPCS.

CONTENTS

I INTRODUCTION AND SUMMARY

Under the sponsorship of the Air Force Rocket Propulsion Laboratory, Stanford Research Institute has undertaken a survey of sensitivity data on NF compounds. The results have been summarized in an SRI final report,[1] a handbook which contains raw, unevaluated data and reviews of some of the structure-sensitivity relationships and thermal decomposition studies. The handbook is to serve as a reference source for investigators engaged in research and development in synthesis and propellant formulations.

This ~~special~~ report is a critical evaluation of several aspects of the problems associated with the sensitivity characteristics of NF compounds. In particular, the report includes: (a) a summary of sensitivity-structure relationships and their significance; (b) a summary of viewpoints expressed by personnel engaged in NF propellant development; and (c) a discussion of sensitivity testing and data reporting. Conclusions and general recommendations are made as they apply to each section.

The accumulation and reporting of the mass of sensitivity data for the final report required a thorough review of sensitivity testing methods which revealed some discrepancies in results reported for various NF compounds. However, trends were discernible in the sensitivity characteristics such as the alkanes, alcohols, esters, and ethers. Thus there was a unique opportunity to have a purview of the whole problem of NF sensitivity. ~~Some~~ of the more objective reviews and correlations that could be made are contained in a discussion section of the final report. It is the purpose of this special report to comment on more subjective aspects of sensitivity problems. We hope to present several suggestions for the safe development and use of NF propellants. In doing this we are drawing upon the knowledge and experience of many people within the explosives and propellant industries. We do not claim that the viewpoints expressed are necessarily original,

1. Stanford Research Institute, Final Report, "Compilation and Review of Data on the Sensitivity and Stability of NF Compounds: A Handbook," AFRPL TR-67-77, March, 1967.

nor do we expect that all suggestions will win acceptance. However, if this document can help to produce action which will results in orderly and accurate research and development in the difficult area of energetic NF materials, it will have served its purpose.

In this report we review the background and significance of the sensitivity characteristics of various classes of NF compounds, the results of our interviews with personnel involved in propellant development, and the problems of testing, with suggestions for improvement in obtaining sensitivity data. In essence we have concluded that the sensitivity properties of NF compounds should be judged in relationship to the stage of development at which the material is being studied. Assuming thermal stability and chemical compatibility are sufficient for further consideration, the compound can probably be manufactured, although it may be very sensitive. In the development stage, a decision as to acceptability should be made not upon the characteristics of the individual compound but upon the sensitivity properties of the propellant composition. Factors influencing the choice of compounds, in relation to sensitivity, are discussed. Standardization of testing methods has apparently been unsuccessful. Problems and possible improvements in obtaining data are discussed; we suggest that a central laboratory should be established for well-instrumented and accurate sensitivity testing of compounds and that only the data obtained from such a laboratory should be used as the basis for decision.

II SENSITIVITY-STRUCTURE RELATIONSHIPS

A. Results from Small-Scale Sensitivity and Thermal Stability Tests

Valid relationships are discernible between the structures of NF compounds and the sensitivity characteristics.[1] Generally, as the energy of the compound increases the sensitivity problem becomes more severe. Correlations between heat of detonation and sensitivity show that compounds which are generally sensitive to the impact test, for example, also have a heat of detonation in the range of 1500 to 1600 cal/gram. For such high energy compounds--particularly for the tris(difluoroamino)methoxy compounds--many other sensitivity characteristics are severe.

Difluoroamino compounds can be divided roughly into three classes: (1) relatively insensitive compounds, substituted with a low ratio of NF_2 groups to carbon; (2) compounds with substitution in a ratio of up to one NF_2 group per carbon, possessing high energy, and exhibiting sensitivity characteristics about the same as those of nitroglycerin or HMX; and (3) ultrasensitive compounds, of very high energy, which are more sensitive than nitroglycerin and HMX and are difficult to desensitize. In the first class are the long chain alkanes, mono and bis substituted with NF_2 groups (exceptions are the geminate substituted compounds, vide infra). In the second class are bis difluoroamino compounds such as the bis(difluoroamino)propanes, tris difluoroamino compounds with a ratio of one NF_2 to one methylene group and substituted in the 1,2,3-, 1,1,2-, or 2,2, positions, and compounds with more than three NF_2 groups. The third class is primarily tris difluoroamino compounds. Much of the desensitization work reported has shown that dilution of an NF compound is the most effective way to reduce its hazard, but the desensitization is sometimes accompanied by excessive loss of energy. Liquid difluoroamino compounds possessing high energy are seldom handled in neat condition and

are most easily handled diluted with solvent. Partial desensitization toward impact or static initiation has been observed by other methods but the treated materials showed extreme sensitivity towards other tests; for example, achieving desensitization to impact did not also achieve desensitization to static initiation.

Sensitivity characteristics of the various classes of NF compounds can be correlated with the number and position of the NF_2 groups on the carbon chain, and some types of substitution are particularly sensitive to certain methods of energy input, e.g., to spark initiation or to friction. The general sensitivity of a compound is established by the NF_2 substitution and is usually not intensified by the effect of other oxidizing groups such as nitro or nitrate.

Sensitivity properties of the alkanes generally reflect the properties which can be expected of other classes such as the alcohols, esters, or ethers. Distinct substitution effects are discernible in the alkanes and can be summarized as follows:

1. Vicinal substituted compounds of a ratio of about two NF_2 groups to three carbon atoms are in the nitroglycerin sensitivity range.
2. Geminate NF_2 groups impart more sensitivity to a compound than does the vicinal group, particularly when the geminate group is terminal, as in the 1,1 position. Desensitization of geminate compounds by increasing the carbon content is ineffective. Generally the energetic geminate compounds are in the nitroglycerin sensitivity range and appear especially susceptible to spark and shock initiation.
3. Separation of NF_2 groups by one or more CH_2 groups appears to make the compounds less sensitive, but the magnitude of change is not large.
4. Increasing the amount of substitution in a carbon chain in a ratio greater than two NF_2 groups to three methylene groups does not increase the ease of initiation.

5. Alkanes, and probably other similarly substituted classes, are susceptible to initiation by low energy input and can propagate by a destructive low velocity detonation wave. Also, these compounds have no practical critical diameter below which detonation will not propagate.

In alcohols there is no discernible effect of the hydroxyl group on sensitivity. However, esters appear to be less sensitive than the parent alcohols, possibly because of a large drop in energy content. Ethers possess the properties of the parent NF substituted hydrocarbon moiety; the energetic compounds are as sensitive as nitroglycerin or more so. In cyclic compounds, alkyl halides, cyanides, and isocyanates there is also no discernible structural effect of the functional groups. The sensitivity of these compounds is equivalent to that of the parent alkanes.

Tris(difluoroamino)methyl and methoxy compounds are in the ultra-sensitive range, being more sensitive than nitroglycerin or HMX and often being extremely sensitive toward any method of energy input. As in the geminate series, diluting the effect of tris(difluoroamino)methoxy group with additional methylene groups does not obviate the effect of the tris methyl substitution. Attempts to desensitize this class of compounds by conventional means were unsuccessful; the compounds were still several orders of magnitude more sensitive than conventional handleable propellant or explosive ingredients.

The thermal stability of NF compounds has been the subject of empirical stability tests and thermal decomposition kinetic studies at several laboratories. Under controlled conditions in which decomposition was caused by purely thermal energy, many NF compounds have shown good thermal stability. (For this discussion, good thermal stability means an indicated lack of decomposition at temperatures up to 100°C for short periods which can be extrapolated to long-term storage conditions.) Homogeneous decomposition studies have shown that C-N bond energy for the C-NF_2 group is about 45-60 kcal, and the NF bond is about 5-10 kcal more stable. Difluoroamino compounds generally decompose by initial unimolecular bond homolysis. Evidence has been obtained for a mechanism of decomposition involving

cleavage of the carbon-nitrogen bond to produce $\cdot NF_2$ radicals as the first step. This first step has been observed for a variety of NF compound classes. Subsequent steps involve an exothermic fluorine migration from the NF_2 group to a carbon radical. If the initial rate-limiting step in an explosion is a unimolecular bond homolysis or an intramolecular migration, little can be done to inhibit the reaction and stabilize the compound. However, certain additives might be effective in trapping the initial products, preventing explosion or avoiding conditions needed for an explosion to occur.

Many difluoroamino compounds are very sensitive to catalytic decomposition under ordinary conditions encountered in the laboratory or in practical use. This is really a reflection of chemical reactivity and not purely a thermal phenomenon, although heat obviously promotes the rate of such reactions. Actually, it appears that many purported thermal decompositions of NF compounds are really reports of results of heterogeneous reactions of the test material catalyzed by container materials such as glass, aluminum, or stainless steel. Hydrogen fluoride elimination from $-CHNF_2$ and $-CH_2NF_2$ groups is common and easily obtainable, as indicated by the activation energy of 15 kcal for HF elimination. In contrast, HF elimination in compounds containing $C-NF_2$ groups is very difficult to obtain under ordinary thermal conditions. The decomposition by HF elimination is autocatalytic and is particularly sensitive to fluoride and water.

B. Sensitivity Characteristics of NF Compounds in Development Studies

We interviewed a number of research workers engaged in NF propellant development in order to obtain views, experiences, and opinions of those engaged closely with multipound quantities of NF compounds.* The objective

*We are indebted to the following people for many very interesting and helpful discussions of NF compound sensitivity problems: Aerojet-General, Sacramento, M.A. Klotz, R.A. Price; Atlantic Research Corporation, James Martin, Lewis Childs, and Joseph Burton; Rohm and Haas Company, John Parrott and Thomas Pratt; Thiokol, Huntsville, Meredith Mather, W.E. Hunter, William Stevens, and Eugene Goree; United Technology Center, Paul Allen and Edward Ives.

was to determine how the sensitivity characteristics of NF compounds affect their manufacture and handling under conditions commensurate with end use.

Several of the general topics discussed covered important aspects of NF compound sensitivity. These included: (a) whether any class of NF compound should not be considered for development because of sensitivity; (b) the probability of achieving sufficient desensitization to use the compounds in propellants; (c) which kinds of hazard, if any, are most critical during development and use; (d) how much additional precaution, compared to ordinary propellant preparation and handling, is necessary for NF ingredients; and (e) if general experience shows that compounds as sensitive as nitroglycerin (or more so) can be utilized in propellant formulations. In the matter of sensitivity testing, discussions primarily concerned the adequacy of present test methods to define hazards in handling and use. The propellant compositions most frequently referred to were the NFPA-TVOPA and PBEP-TVOPA binder-plasticizer combinations. Other plasticizers included OPE and HPE for comparison with TVOPA, and solids briefly utilized as oxidizers were BTU and INFO-635.

Several areas of concurrence were apparent in the opinions independently expressed by the laboratories; none was aware of the viewpoints of others. There was general optimism about successfully manufacturing, handling, and using NF polymers and high energy plasticizers. Techniques have been developed for remote manufacture and for handling the products in solution. Neat materials, especially the plasticizers, were usually not handled. All ingredients were thus manufactured, transported, and formulated in solution and the solvent was stripped off before final curing under remote control. Although the NF propellants prepared in this way were sensitive by several tests they were less sensitive than the ingredients individually, and no more sensitive than presently developed propellants containing HMX and other "normal" ingredients. It was felt that any NF material which cannot be handled in this manner should be avoided. Sensitivity characteristics of the individual ingredients were not considered important to a decision to accept or reject a material, but it was believed that the properties of the final propellant should be the determining factor.

There was general reluctance to consider solid NF oxidizers seriously, particularly the solid perfluoroguanidine derivatives. Great concern was expressed about the static and friction sensitivity of these materials. Friction initiation could come about from shearing action during mixing, from solid-solid or solid and mixer blade rubbing. Static discharge was considered hard to prevent in an absolute sense or to guard against sufficiently to provide a reasonable margin of safety for NF solids. INFO-635 was an ultrahazardous material which represented an extreme problem several orders of magnitude too sensitive to handle with comparative safety.

Of the tests presently in use, most were considered useful but confusing and could be better. The greatest need seems to be for meaningful static and friction sensitivity tests, because it was felt that energy applied by friction and static was more insidious and more difficult to prevent or obviate. The need for better standardization of tests was discussed repeatedly as was the need for better correlation of tests with actual handling conditions. Some test data are obtained but are not taken seriously--at least, the results are not the entire basis for decisions on whether to expose personnel to the materials. Decisions on how best to handle a material are arrived at on the basis of a considered judgment of overall handling experience, including the experiences of personnel involved in studying the material on a scale ranging from one gram to several pounds.

Below are summaries of various statements not generally repeated by other laboratories; they are helpful in judging how to produce and use NF materials.

1. Rohm and Haas tests the hazards of the manufacturing process as well as the ingredients; all solutions and mixtures involved in the preparations are tested, as well as the pure NF materials. At each stage of development, sensitivity testing is repeated, the results are reviewed, and decisions are made on the degree of manual handling allowable; handling procedure is always subject to frequent sensitivity testing.

2. Aerojet has found a rough correlation between handling hazard and explosiveness by using the differential thermal stability test. They feel that a material which explodes rather than burns from thermal excitation is generally very hazardous; INFO-635 is an example.
3. Atlantic Research Corporation compared a TVOPA-NFPA mixture with TMETN by impact, friction, and gap tests and found the former less sensitive by these tests; TMETN was received undiluted in 55-gallon drums. Nonetheless, the NF compounds are being handled warily.
4. Rohm and Haas by its tests found that during various stages of processing Plastisol tested more sensitive than some NF compounds during processing. The final Plastisol was in the Class 2 hazard category.
5. United Technology Center observed an increase in sensitivity of NF compositions when hydroxylammonium perchlorate and aluminum hydride were introduced. This group felt that safe production and use of NF materials were partly a matter of engineering for automated remote processing and partly an education process for the personnel involved in knowing the limitations of the materials being prepared.

C. Conclusions and Recommendations

The sensitivity characteristics of pure, high energy NF compounds indicate that they are among the most sensitive of propellant ingredients and generally equivalent to nitroglycerin and HMX in sensitivity properties. Nonetheless, recent formulation studies at many laboratories have demonstrated that some of the most sensitive of the compounds can be manufactured, formulated, and handled in the final composite form. Hazards to be considered arise primarily in two areas--manufacture and formulation, and handling and use of the propellant composition. Consequently, in judging the usefulness of a compound two sets of sensitivity measurements should be run. The first of these, of course, are tests on the pure materials during research, characterization, and scale-up. However, before application to studies in

formulation a compound must have thermal stability and must be compatible with other ingredients and the environment. Without meeting these criteria (the standards of thermal stability and compatibility must be set realistically) it is useless to consider a compound for further study. The second set of tests should be run on small-scale, trial propellant formulations and these data should be used, apropos sensitivity, as a basis of decisions for further study. Thus the crux of the whole matter is the severity of the sensitivity problem in the final propellant formulation. Since successful manufacture and handling of ingredients in solution has been well demonstrated, the severe sensitivity properties of an individual compound can be obviated. The NF compounds most amenable to handling in solution are the liquids and polymers that have moderate sensitivity properties.

This conclusion does not infer that any compound, no matter how sensitive, should be manufactured and formulated. Three factors are interrelated in arriving at a decision: one, nonexcessive cost of preparing and guarding against acidents from a very sensitive compound; a second, the relative lower cost of achieving safety of one ingredient compared with the cost incurred for another compound of similar energy; and the third, the amount of increased performance gained in using a sensitive material in a formulation over that of present systems (many of which are themselves rather sensitive by many tests). Compounds which may have difficulty meeting all these criteria as well as sensitivity standards are the NF solids and PFG derivatives, the former because of demonstrated static and friction hazard and the latter because of the possibility of achieving the same energy advantage from other compounds more amenable to handling.

Frequently the question has arisen of how severe the sensitivity problem has to be before a compound or propellant composition is rejected for field use. There appears to be little purposeful guidance by government agencies on this point. We found many who were working on propellants who had too little information on mission requirements, a propos sensitivity, and on the degree of acceptable sensitivity. It would be very helpful for the military agencies, project managers and technical contractual officers to publish the sensitivity acceptance criteria to be met by a composition before field use. Such a document should help supervisors on the bench level to avoid some obviously unfruitful research and development effort.

III PROBLEMS OF TESTING AND DATA REPORTING

A. Lack of Interlaboratory Correlation of Test Results

During the accumulation and tabulation of reported sensitivity data, it became apparent that there was a lack of agreement and correlation of test results among laboratories. On a few occasions, reported impact sensitivities indicated both insensitivity and extreme hazard. Inconsistencies were also revealed in the values reported from other test methods. Consequently, quantitative values could not be applied to the sensitivity of a material toward a particular test, a fact which forced a qualitative review of trends and general characteristics.

Several kinds of tests are being used to evaluate sensitivity, a property which has no single quantitative term; instead, the sensitivity of a compound can only be accurately described in relation to the type of stimulus applied. Generally, five types of tests are used--impact, shock, friction, static, and thermal. The standard tests recommended for use in each category have been modified and remodified, in many instances seemingly without control. The consequence of this "fragmentation" of test methods is that we now have a multiplicity of tests creating a confusing mass of data, much of which has no meaning beyond the immediate confines of the testing laboratory. However, we must point out that there has always been a sincere effort to define the hazard of a particular compound or system for immediate or large-scale handling and use.

Many laboratories realized the qualitative aspect of the results being obtained and consequently tried to relate their results to known liquid and solid propellant and explosive compounds. The known handling characteristics of the standard compounds, then, by inference allow an anticipation of handling problems with the test compound. Reporting relative sensitivity data is useful. However, many laboratories compared liquid compound test results with solid standards, particularly for the impact test. The results of this test and others are especially influenced by the physical state of the material, and the test values of a liquid compared with those of a solid have no real meaning.

Sometimes it was difficult to be sure that the test result being reported had been obtained on pure materials. Impurities drastically affect sensitivity results from some tests, and we found little evidence in many instances that purity control was attempted or that it was realized to be important. Thermal stability tests were often particularly suspect because of the lack of purity control.

The general sensitivity properties of many compounds are obscure because of incomplete testing. In many instances friction and static sensitivity testing would have been desirable. Personnel engaged in development of NF systems felt that friction and static sensitivity testing as now done is inadequate. There is little correlation between sensitivity testing and handling conditions per se, at least in a quantitative sense. Generally, an estimate of actual hazards to be encountered in a practical situation was always obtained subjectively by development personnel on the basis of prior experience and qualitative empirical testing.

The accumulation of thermal stability data for the final report revealed that a variety of tests was being used. A lack of standardization in stability testing also permitted a melange of methods to be used, none of which could be correlated. Nor were we able to find a study of the correlation between long-term storage and the accelerated thermal stability tests being used. The answer to the question of how thermally stable a compound or propellant needs to be for acceptance seems obscure. Unfortunately, it appeared that many laboratories used a surveillance temperature which was not too hard on their compound or propellant. There is great danger in incorrectly handled stability measurements because an improperly run test may not reveal an autocatalytic decomposition process which could later show up as a spontaneous explosion in the propellant.

B. Deficiencies in Current Testing Procedures

Corollary aspects of sensitivity are undetected by empirical testing. These include the real failure diameter, susceptibility to low energy input, ability to propagate, possibility of damaging low velocity explosion waves, definitive effect of porosity and physical state, and action in bulk versus

action in small test quantities (lack of scale-up). Without knowledge of these characteristics, one is forced to treat all materials as very sensitive. The empirical tests consequently have been of limited value for predicting situations beyond the scale of the tests.

The reasons for lack of correlation may often be that the tests are concerned with different events in the list above, i.e., different paths of decomposition, and that the tests do not reflect the kinetic aspect of the test method, inasmuch as the rates of stimulus input are different in each case. A card-gap test purports to measure sensitivity to detonation but more often may give numbers which reflect the materials of test apparatus and the procedure used in the test. In one instance, research showed that the gap test as run presently is invalid for liquids such as difluoroamino propanes. Results are dependent upon the geometry of the system and the materials of construction, with initiation occurring at the walls of the container. Thus the test result did not accurately reflect a property of the material but rather the way the experiment was designed.

In another instance involving the same compounds, low velocity waves of considerable destructive power were detected at small critical diameters. Such low velocity wave phenomena cannot be detected accurately by present test methods; the closest observation is perhaps that "something not quite the same happened." From the safety standpoint, low velocity detonation (LVD) is a serious matter because such waves are destructive. In some cases a relatively low amount of energy input caused ignition and propagation. The fact that LVD waves will travel in small diameters dictates that NF liquids will have to be handled in solution.

C. Conclusions and Recommendations

It is problematical whether the plethora of sensitivity tests can be reduced to a manageable number of well-standardized procedures. It appears that modifying a standard test is so common as to be inevitable. This situation may have arisen because of a general feeling that current tests do not really indicate handling hazard. However, a well-enforced minimum series of standard tests should be attempted.

Research on test methods seems to be necessary to determine whether the test measures what it is claimed to measure, and to investigate the relationship of a particular test with actual hazards encountered and not with the "science" of testing. It would help also if the handling hazards were enumerated, defined, and advertised so that research in testing would have a development objective. Particularly it seems very desirable to develop a useful indicative static and friction sensitivity testing procedure.

Research with well-instrumented procedures should continue in defining structure-sensitivity relationships on compounds chosen to represent changes in only one aspect of the structure; examples are the alkane difluoroamino isomers or a consistent series of alchols, esters, ethers, or nitro compounds. The basic phenomena of initiation and propagation still need study, especially a definition of the extent of low velocity detonation. Thermal decomposition studies are needed for further insight into the chemical mechanisms of initiation and propagation of explosion and for developing our knowledge of how successful desensitization affects the chemical mechanism of decomposition or explosion.

Finally, in order to keep sensitivity testing under control, we suggest that a central laboratory should be established for well-instrumented and accurate sensitivity testing of compounds and that only the data obtained from such a laboratory should be used as the basis for decision.

GLOSSARY

NFPA	2,3-Bis(difluoroamino)propyl acrylate
TVOPA	N_2F_4 adduct of 1,2,3-tris(vinoxy)propane
OPE	1,2-Bis(difluoroamino)ethyleneglycol-2,2,3,2′,2′,3′-hexakis(difluoroamino)dipropyl ether
HPE	1,2,3,1′,2′,3′-hexakis(difluoroamino)propyl ether
BTU	Di-N,N′-[tris(difluoroamino)methyl]urea
INFO-635	2-[tris(difluoroamino)methoxy]ethylamine perchlorate
TMETN	Trimethylol ethane trinitrate
PFG	Perfluoroguanidine

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13. ABSTRACT

This report presents a general review of the sensitivity characteristics of NF compounds and their significance. It includes a discussion of sensitivity-structure relationships, a summary of viewpoints on sensitivity expressed by personnel in NF propellant development, and a discussion of sensitivity testing and data reporting. Conclusions and recommendations are made. (Unclassified)

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